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AN IN-SITU CYCLIC STRESS EXPERIMENT AT THE CLINTON P. ANDERSON MESON PHYSICS FACILITY (LAMPF)† FOR DETERMINING THE EFFECT OF DISLOCATION VIBRATION ON VOID GROWTH IN METALS DURING IRRADIATION

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We report our experience with the first in-situ cyclic-stress irradiation at LAMPF. An proton beam ion current of 3-6 μA of 800 MeV protons was utilized for 24 days irradiation

Earlier, radiation damage effects of 800 MeV protons incident on a 1-cm thick Cu target were calculated by using the nucleon-meson transport code to determine the nuclear reactions produced by the protons, and the theory of Lindhard et al. to evaluate the resultant damage energy deposited in the target. These calculations have now been extended to Al. Damage effects were found to be nearly uniform through a 1-cm target thickness, and the results obtained can be expressed in cross section form. The calculation yielded a damage energy cross section of about 63 barn-keV, a nuclear transmutation cross section of 0.44 barns, and indicated copious hydrogen, helium and neutron production.

An analysis of the effect of dislocation vibration on the efficiency of a dislocation line as a sink for point defects predicted that dislocation vibration should suppress void growth. The effect results from the fact that the dislocation will sweep up vacancies, which diffuse less rapidly than interstitials. The experiment is intended to test this theory.

The growth rate of voids in Al under simultaneous proton irradiation and cyclic stressing are compared to that of samples irradiated at the same time but without any stressing. The samples are placed one behind the other along the proton path so that identical irradiation histories can be achieved. The temperature of the samples is controlled, known and uniform. The initial preirradiation state is a prestrained state of a few hundred stress cycles. The samples are irradiated without stress through the "incubation" period for void nucleation before the cyclic stress is applied.

1. INTRODUCTION

Under irradiation, in the void growth temperature domain, the vacancy concentration becomes orders of magnitude greater than the thermal equilibrium value. This then constitutes the large force driving a vacancy flux to nucleated voids, leading to void growth, swelling, and embrittlement. If dislocations can be made a stronger sink for vacancies, for example by vibrating the dislocations, relative to and in competition with the voids then the void growth rate should be less. An analysis [1] by Weertman and Green was made of the effect of vibrating edge dislocations on void growth in metals. Increasing the dislocation sink strength is manifested by the "sweeping up" effect of moving edge dislocations. The experiment reported herein takes advantage of the high penetrating power of 800 MeV protons and places samples, one with an imposed cyclic stress, the other with no stress, in line along the proton beam to assure identical irradiation histories in both samples (Fig. 1), given that multiple

SAMPLE ARRANGEMENT

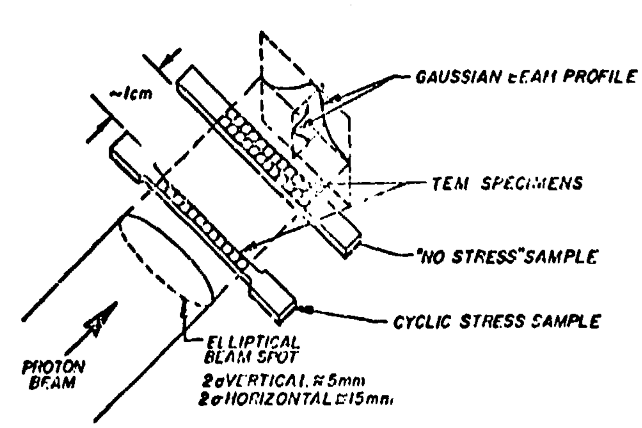

Fig. 1

scattering effects are accounted for. An analysis of the flux effect of multiple scattering on aligned foils was made [2] and indicates an insignificant effect for the geometry chosen here.

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The Clinton P. Anderson Meson Physics Facility (LAMPF) accelerates protons to 800 MeV. The analysis of Coulter, et al (3) calculated the damage and transmutations that would occur in Cu under 800 MeV proton bombardment. This work has now been extended to other metals including Al [4]. For Al, the damage energy cross section was found to be 63 barn-keV, and since the threshold displacement energy is 17 keV, the damage rate is

$$\frac{\text{dpa}}{\mu\text{A}}\frac{\text{cm}^2}{\text{hr}} = 3.3 \times 10^{-5} \qquad (1)$$

where dpa is displacements per atom. The gas generation rates were calculated to be 300-400 ppm/dpa for hydrogen and 70 ppm/dpa for helium.

The LAMPF accelerator operates at 120 Hz, with an "on time" of 6%, i.e., 0.5 ms on every 8.33 ms. A recent analysis [5] indicated that this pulse structure would not alter the void growth rate relative to steady irradiation if temperature is maintained equal for the two cases. The temperature-time history of an Al foil under 800 MeV proton beam with a Gaussian beamspot intensity distribution was calculated [6]. An insignificant (< 1 C°) temperature rise was calculated for the sample centerline for our targeting conditions.

A beam of 5×10^{18} protons $m^{-2}s^{-1}$ current density is available at the "Nuclear Chemistry In-Air Targeting Station" in Area B at LAMPF. This current density produces a damage rate, in Al, of approximately 0.003 dpa day^{-1} (This beam would produce about a 10 x greater damage rate in a high Z material such as Mo or W.) Targets are mounted in a target box of about 0.5 m^{-3} volume and are serviced via a walk-thru maze and umbilicals which terminate in a control trailer (Fig. 2).

High purity Al metal exhibits low fluence void growth [7] at 328 K. Although other locations at LAMPF would yield much higher dpa rates, our choice was a compromise between achievable fluence, ease of temperature control, facility availability, and impact on LAMPF. Our final samples were fabricated from the same high purity Al as used in the studies reported by Stiegler [7].

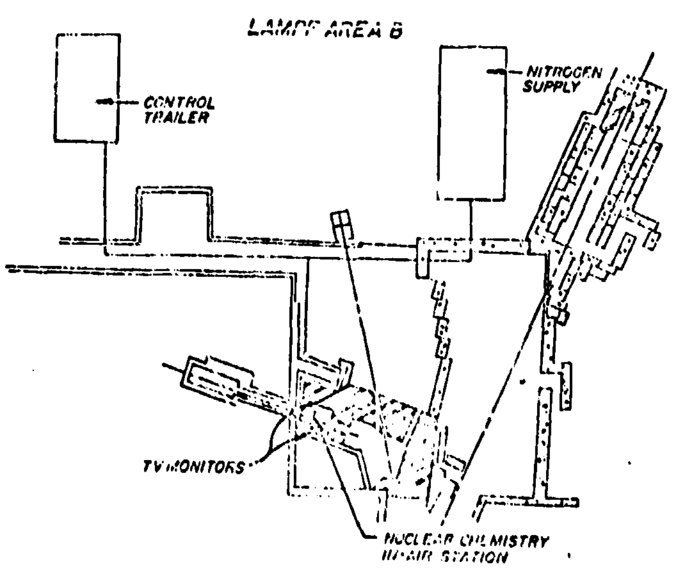

Fig. 2

LAMPF is a national facility where candidate experiments are reviewed. The cyclic-stress experiment was awarded 24 days of prime beam time after review.

At this writing, approximately 24 days of irradiation have just been completed. The samples are undergoing radioactive decay to a lower level before optical and electron microscopy work commences.

2. REQUIRED CONDITIONS

The distance λ a dislocation glides under stress must be made greater than the characteristic diffusion distance, d,

$$d = \left(\frac{D}{4\nu}\right)^{1/2} \qquad (2)$$

where ν is the frequency of the stress and D is the vacancy diffusion coefficient. The glide distance λ for a material of modulus μ depends on the distance ℓ between pinning points according to the expression [1]

$$\lambda = b\left(\frac{\tau}{\mu}\right)\left(\frac{\ell}{b}\right)^2 \qquad (3)$$

The distance between pinning points ℓ under irradiation is unknown and will be a function of the initial dislocation state, the fluence, the temperature and the strength of the pinning points, but we assume that a reasonable value is $100\,b < \ell < 1000b$. Out-of-irradiation cyclic testing on our samples at 50°C established a "safe" cyclic stress value of $\tau/\mu = 1.35 \times 10^{-4}$. Then $1.4b < \lambda < 140b$. Calculated characteristic diffusion distances are as shown in Table 1.

Table 1
Characteristic Diffusion Distances in Aluminum

Temperature (°C)	D vacancy (m^2s^{-1})	d (b) 1 Hz	10 Hz	100 Hz	1000 Hz
20	2.1×10^{-11}	7	2	λ	
50	7.5×10^{-10}	50	13	5	1
85	6.6×10^{-9}	140	43	14	5

Our desired effect would be most pronounced at the lower temperature since then λ would exceed d.

A temperature of 50°C was chosen as a compromise between enhancing the effect by lowering the temperature while maintaining a level where sufficient void growth would occur during the proposed irradiation. A more detailed analysis of the magnitude of the effect of dislocation vibration on growth has been made and will be compared to the measured effect later.

3. EXPERIMENTAL SYSTEM

A remote, tension-tension, irradiation device was designed, built, developed and debugged (Fig. 3). It incorporated a temperature control system (Fig. 4). Dead weight loading was used for reliability reasons. The load train included a 0.4 mm diameter flexible cable passing

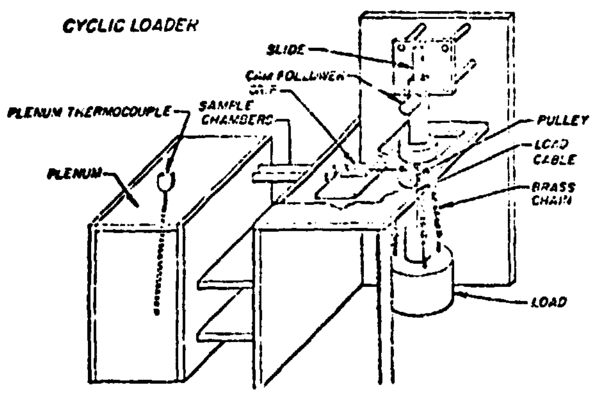

Fig. 3

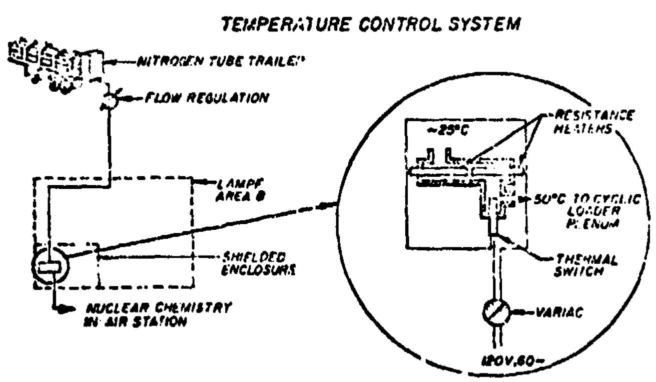

Fig. 4

over a pulley, and a load pan. The load was raised and lowered via a cam, a captured slide, and a constant speed motor. The link between the slide and load was a chain to disallow overstressing. A spring dampens the stress amplitude at the instant the load is transferred from the chain to the sample.

To achieve temperature control, stored compressed nitrogen was used for reliability. The gas flowed through a service umbilical to a heater system near the irradiation device (Fig. 4). The gas flow rates and monitoring procedures were developed in bench top experiments. A shielded thermocouple in the gas plenum was outside the region of significant beam heating and radiation and therefore was taken to be a reliable monitor. The control system maintained the gas temperature at 50°C to within 1/2 C°.

Alignment of the samples to the beam was required at the start of the irradiation. Although the beam in Area B at LAMPF is very stable in spacial alignment, some drift did occur. Alignment was monitored with a closed circuit television system that viewed a "phosphor screen" in the proton beam directly behind our experiment. Fine alignment was accomplished by steering the beam magnetically into a thermocouple in line with the samples. The output of this thermocouple as well as the N_2 gas temperature were continuously recorded.

High purity Al was chosen as the testing material for the following reasons: (1) voids form in Al at low dpa levels; (2) the swelling temperature range for Al is easily achieved, and (3) its activation is low for the desired dose. Samples were cut with a wire saw from 99.9999% pure Al obtained from a commercial source. Samples were then rolled, packed within mylar, to the final specimen thickness of 0.25 mm. Purity was confirmed by microprobe analysis. After pickling in a solution of 20 g NaOH-100ml H_2O samples were milled between mylar, repickled, and finally annealed for 1 h at 200°C in a 10^{-7} torr vacuum.

Cyclic stress testing between 0.7 σ_{yield} to 0.2 σ_{yield} established that the time to rupture would be more than the irradiation time at a stress level of 0.3 σ_{yield} or less. A cyclic loading frequency of approximately 0.3 Hz was utilized. Surface microscopy on the cyclic stressed unirradiated samples showed closely spaced slip bands, and a large grain size. Our final operating parameters are noted in Table 2.

Table 2
Operating Conditions for the Irradiation

Cycle time	=	0.3 Hz
Temperature	=	50°C
Load	$\cong$	0.3 σ_{yield}
Damage rate	=	0.003 dpa·day^{-1}
Total time	=	590 h

A remote control trailer serviced the experiment (Fig. 2). Strip chart recorders monitored beam alignment, gas temperature, as well as LVDT motion of the sample grip. No problems with signal control and monitoring were experienced.

4. IRRADIATION EXPERIENCE AND CONCLUSION

Because the irradiation source is a beam, activation of components is less than would occur with a larger source of radiation, such as the interior of a reactor. Because of this, it was feasible and practical to maintain the experiment hands on, after 10 hours of radioactive decay. All in-situ experiments are difficult from a reliability standpoint, and periodic maintenance by us was required in our experiment. Maintenance days are scheduled at LAMPF every 14 days; additional entries can be requested and affected when necessary. The experiment could not have been successfully completed without the maintenance time. For example, the closed circuit TV system that observed the loading and unloading required replacement after each 5 days of use due to radiation effects.

We conclude that controlled, in-situ, mechanical radiation damage experiments can be done under irradiation by 800 MeV proton accelerator beams. Since a "beam" does not activate "peripheral" components to an unreasonable level, the experiment can be serviced "hands-on." We feel that this service capability is a large advantage over in-reactor experimentation.

We have utilized available nuclear data to calculate displacement damage energy, beam heating, and multiple scattering effects. Post irradiation analysis will help to qualify our calculations. This calculational capability and the availability of a large "in air" experiment station with "hands on" access provides an excellent environment for future, difficult in-situ mechanical tests. An additional experiment, similar to the one reported herein, in which cyclic stress and concurrent radiation would be applied continuously throughout the irradiation, is being planned to test the possible effect of dislocation vibration on void nucleation. Also, future work will be directed at establishing how irradiation of metal under stress modifies its time stress and temperature dependent response to that stress.

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